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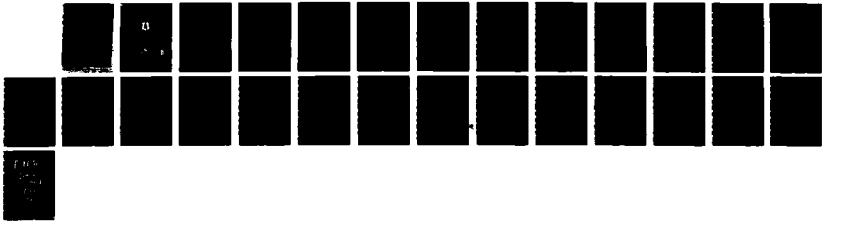

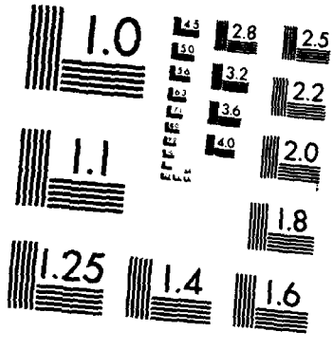

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FOREIGN TECHNOLOGY DIVISION

ACRYLATES AND METHACRYLATES

by

L.A. Morozov

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PARTIALLY EDITED MACHINE TRANSLATION

FTD-ID(RS)T-0936-87 15 September 1987

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ACRYLATES AND METHACRYLATES

By: L.A. Morozov

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Foreign page numbers occur in the English text and may be found anywhere along the left margin of the page as in this example:

> In them occurs the state named "night blindness" - hemeralopia, which, according to the current point of view, is a result of damage of the rod-shaped apparatus of the eye.
>
> Page 51.
>
> However, in recent years it has been shown that with the hereditary pigment degenerations in animals the biochemical changes are observed in all celluar elements of the retina.

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Block	Italic	Transliteration	Block	Italic	Transliteration
А а	*А а*	A, a	Р р	*Р р*	R, r
Б б	*Б б*	B, b	С с	*С с*	S, s
В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
Е е	*Е е*	Ye, ye; E, e*	Х х	*Х х*	Kh, kh
Ж ж	*Ж ж*	Zh, zh	Ц ц	*Ц ц*	Ts, ts
З з	*З з*	Z, z	Ч ч	*Ч ч*	Ch, ch
И и	*И и*	I, i	Ш ш	*Ш ш*	Sh, sh
Й й	*Й й*	Y, y	Щ щ	*Щ щ*	Shch, shch
К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

*ye initially, after vowels, and after ъ, ь; e elsewhere.
When written as ё in Russian, transliterate as yё or ё.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	$\sinh^{-1}$
cos	cos	ch	cosh	arc ch	$\cosh^{-1}$
tg	tan	th	tanh	arc th	$\tanh^{-1}$
ctg	cot	cth	coth	arc cth	$\coth^{-1}$
sec	sec	sch	sech	arc sch	sech^{-1}
cosec	csc	csch	csch	arc csch	csch^{-1}

Russian	English
rot	curl
lg	log

GRAPHICS DISCLAIMER

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ACRYLATES AND METHACRYLATES.

L. A. Morozov.

Among monomers utilized in production of polymeric materials, an important place is occupied by acrylic and methacrylic acids and especially their esters. Acrylic and methacrylic monomers can be polymerized and copolymerized with other vinyl monomers by all known methods with the formation of different high-molecular compounds.

These polymers and copolymers possess important properties: by significant weather resistance, transparency, resistance to aging, elasticity, water resistance, heat resistance, high mechanical and dielectric characteristics, good adhesive ability, etc. Acrylates and methacrylates found use in all branches of national economy. At present the universal production/consumption of acrylates achieved wide scales and are approximately 650-700 thousand t per annum. However, the increasing demand for these polymeric materials is still restrained by the high cost/value of initial monomers. Therefore a question about the development of the new economical methods of obtaining the acrylic derivatives from the accessible and mass raw material is extremely urgent.

ACRYLATES.

Since beginning of organization of industrial production of acrylates it passed a little more than 30 years, and for this small interval of time original diagram of ethylene-cyanohydrin process underwent significant changes. The more economical methods of the synthesis of raw material and acrylic monomers were found, which contributed to reduction in the cost/value of these monomers and to expansion of the market for sale.

For the first time acrylic acid was obtained in 1850 by oxidizing acrolein by atmospheric oxygen. A little later the methyl and ethyl acrylic esters were synthesized from the appropriate esters of α-dibromopropionic acid.

In the Soviet Union for the development of the method of production of acrylates the synthesis of methylacrylate began in 1948. For the first time industrial standards of methylacrylate, synthesized from ethylene cyanohydrin according to periodic diagram, were obtained in 1953.

At present acrylates occupy visible place among contemporary polymeric materials and are found new fields of application. The emulsions, utilized in the tanning industry as the basic primer for the nitrocellulose coatings of skin, were one of the first applications of acrylic polymers. Subsequently, emulsions of acrylic

polymers began to be used as components for the system of water/aqueous coatings.

In textile industry emulsions on basis of acrylic polymers are used as bonding agents for bonded materials, basis of upholstering cloths, connecting/cementing agents for pigments, utilized for staining/coloring and gasket/filling of cloths and for impregnation.

In paper industry acrylic emulsions are used as transparent, colorless coatings, stable to solvents, to oils. The paper, covered with acrylic polymers, possesses a good gloss, flexibility and by the capacity to accept printing ink.

The paint and varnish industry, which produces acrylate paints/colors on basis of copolymers of acrylates with styrene and vinyl acetate and paints/colors, which consist of acrylates, occupies first place on consumption of acrylic emulsions. A rapid increase in the consumption of acrylate emulsions with the paint and varnish industry is connected with the output of the water-diluted paints/colors, utilized both for the internal, and for external coloring of assignments, all possible constructions/designs, articles made of the cement, tree/wood, etc. Acrylic emulsions are used for the enamels, used for coating of cables, as the protective coatings, which preserve the color of the articles, and also for the polishihg of floors. The acrylic natural rubbers, which are obtained by the emulsion copolymerization of butylacrylate with acrylonitrile, are

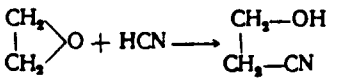
very resistant to the heat aging.

Wide acceptance as coagulant and highly efficient cross-linking agent of soil was obtained by polyacrylamide.

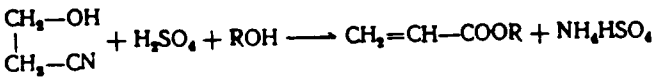
Furthermore, polyacrylamide is used for preparing flocculants for accelerated process of settling/standing into filtrations in chemical and other branches of industry.

Industrial methods of synthesis of acrylates. In the industry acrylates are obtained on one of the following methods:

- from oxide of ethylene and hydrocyanic acid through ethylene cyanohydrin. In the presence of basic catalysts ethylene oxide easily adds hydrocyanic acid with the formation of ethylene cyanohydrin:



Dehydration of ethylene cyanohydrin leads to obtaining of acrylonitrile. Ethylene cyanohydrin can be hydrolized and etherified for one operation, as a result of which is obtained acrylic acid or its ethers/esters:



This method at present is used for industrial obtaining of acrylic acid and acrylates in the USSR:

- from acetylene and carbon monoxide. It is known that carbon

monoxide can be connected to acetylene with the formation of acrylic acid and its ethers/esters:

$$C_2H_2 + CO + H_2O \longrightarrow CH_2{=}CH{-}COOH$$

Gaseous oxide or carbonyl of nickel serves as the source of carbon monoxide. Process is conducted continuously at 40-42°C and normal pressure. The yield of acrylates comprises 80-90%, taking into account acetylene and entire carbon monoxide.

- from ketene and formaldehyde through β-propiolactone.

Condensation of ketene with formaldehyde flows/occurs at room temperature in presence of catalyst (for example, aluminum chloride) with formation of β-propiolactone:

$$CH_2{=}C{=}O + CH_2O \xrightarrow{AlCl_3} \begin{matrix} CH_2{-}C{=}O \\ | \quad | \\ CH_2{-}O \end{matrix}$$

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The β-Propiolactone reacts with alcohols, being converted into acrylic esters:

$$\begin{matrix} CH_2{-}C{=}O \\ | \quad | \\ CH_2{-}O \end{matrix} + ROH \longrightarrow CH_2{=}CH{-}COOR + H_2O$$

The method finds industrial use in the presence of cheap acetone or acetic acid, by pyrolysis of which ketene is obtained.

- from acrylonitrile according to reaction:

$$CH_2{=}CH{-}CN + H_2SO_4 + ROH \longrightarrow$$
$$\longrightarrow CH_2{=}CH{-}COOR + NH_4HSO_4$$

At present are conducted searches for new syntheses of acrylates, whose realization must make possible to produce even cheaper acrylic monomers. Thus, is known work on the oxidation of propylene by nitrogen oxides in the nitric acid; in this case predominantly is formed α-hydroxypropionic acid, as a result of dehydration of which is obtained acrylic acid. The method of the two-stage oxidation of propylene by oxygen into the acrolein and the latter into acrylic acid is studied.

Acrylic acid can be obtained directly from propylene by its atmospheric oxidation in presence of solid catalysts without liberation/precipitation of acrolein.

In principle acrylic acid can be obtained into one stage. From all the existing methods of obtaining of acrylic acid and its ethers/esters the single-stage method is most promising, since in it natural or petrochemical raw material directly is used and virtually in the process there are no production wastes and waste water.

Single-stage process of paraphase oxidation of propylene by atmospheric oxygen on solid catalyst is developed/processed also in Soviet Union. In 1965 there was created the installation on which the technological parameters of the node of oxidation are depleted and the diagram of the liberation/precipitation of acrylic acid is mastered. In the table the tentative data about the shop prime cost of 1 t of acrylic acid of the acid, obtained by different methods, are cited.

As can be seen from table, cost/value of acrylic acid, obtained by single-stage method of oxidizing propylene, two times lower than cost/value of acid, which is developed by other methods. According to the five-year plan the developments of the national economy of power on acrylates must increase 2 times. In connection with this especially valuable is the development the Soviet chemists of the single-stage method of obtaining acrylic acid.

Shop prime cost 1 t of acrylic acid, obtained by different methods.

(1) Статьи расхода	(2) Стоимость, руб. (3) Методы получения		
	(4) через этиленциангидрин	(5) через акрилонитрил	(6) окисление пропилена
Сырье, полуфабрикаты за вычетом используемых отходов (7)	488—76	420—99	111—95
Энергетические затраты (8)	31—53	26—59	91—20
Амортизация (9)	14—87	14—87	28—96
Зарплата с начислениями (10)	15—36	15—36	3—50
Цеховые расходы (11)	25—10	25—10	31—42
Цеховая себестоимость (12)	575—62	502—91	266—13

Key: (1). Articles of expenditure/consumption. (2). Cost/value, rub. (3). Methods of obtaining. (4). through ethylene cyanohydrin. (5). through acrylonitrile. (6). oxidation of propylene. (7). Raw material, semi-finished products minus utilized withdrawals/departures. (8). Power expenditures. (9). Amortization. (10). Wage with additions. (11). Shop expenditures/consumptions. (12). Shop prime cost.

Methacrylates.

Among contemporary polymeric materials polymethacrylates and copolymers on basis of methacrylic monomers are separated/liberated. The great use in methacrylates and predicted rapid increase in their production are conditioned on the development of different areas of contemporary technology, which provide the application of polymeric materials, which possess high performance properties. A number of these properties, first of all, includes: the heat resistance, water resistance, weather resistance, aging resistance, effect of fuels,

high mechanical and dielectric characteristics, good adhesive ability, radiotransparency, etc. Methacrylates satisfy most fully these requirements; moreover, the obtaining of methacrylates is secured with accessible Soviet raw material.

Since the beginning of industrial production of methacrylates about 30 years passed. Initially methacrylates were obtained from cyanohydrin through the stage of α-oxyisobutyric acid. At the same time it was shown that in the technical sense not are less important ethers/esters of methacrylic acid, which initially were obtained from the α-hydroxyisobutyric acid.

Production of methyl methacryate and organic glass on industrial scales in USSR was organized in 1936. During three subsequent decades this process underwent significant changes. Methacrylates began to be obtained directly from acetone cyanohydrin and monohydrate of sulfuric acid, which considerably reduced the cost them and spread the fields of application. From methacryates there are obtained high-molecular polymers, which possess greater hardness and higher softening temperature, than the polymers of acrylates.

Initially methacrylates were used as safety glass interlayer, and more lately methyl ether of methacrylic acid began to be used for production of organic glass, which possesses high transparency, by weather resistance and by strength. On its qualities organic glass satisfies the requirements of contemporary aviation for the materials,

used for glazing of aircraft. At present there are developed formulas of the sufficiently large assortment of decorative and illumination engineering block organic glasses, painted, mainly, by organic dyes/pigments.

These glass can be divided into following groups:

- transparent painted.

- smoked and black.

- turbid not painted.

- turbid painted.

- organic glasses with a textured surface.

Coloring organic glass is realized in polymerization. The best results during dyeing/coloring of polymethyl methacrylate are achieved/reached by applying the fat-soluble dyes/pigments. To a lesser degree dispersed and alcoholsoluble dyes/pigments are suitable. Transparent painted organic glasses are obtained the violet, blue, azure, green, yellow, orange, and red colors of different intensity of coloring and most different hues.

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Transparent painted organic glasses are used as the light filters for the visible region of the spectrum in the radio engineering, aviation, motor vehicle industry and in the medicine.

Formulas of light resistant smoked and black organic glasses with application of highly dispersed gas black are developed.

Turbid unpainted and painted organic glasses are prepared. On the density clouding glass is subdivided into 4 groups. These glass are used as the scatterer for the illuminating lamps with the fluorescent lamps and the incandescent lamps in the illumination engineering industry. For decorative purposes, to the surface of glass different designs and ornaments will be brought in.

In food, chemical and other branches of industry ducts, prepared from organic glass, found wide application. On them it is possible to transport the aqueous solutions of weak acids and alkalis. Ducts of organic glass are more strong/durable in the comparison with the ducts from the silica glass.

Role of organic glass in contemporary technology continuously increases/grows, so that to give being all-inclusive analysis of its application is virtually impossible.

Wide application found slurry polymethyl methacrylate for press casting and extrusions. This material on the strength and to transparency is not inferior to block polymethyl methacrylate. From the very beginning these materials found use in the illuminating, motor vehicle industry, in electrical engineering and other fields.

Latexes - methacrylates are used for obtaining copolymers with monomers of acrylic series/number. Emulsions on the basis of the copolymers of methacrylates and acrylates are used for the same purposes, about which there was discussion in the section devoted to acrylates. Polymethyl methacrylates are used also as the ash-free additives to petroleum and synthetic oils, for the thickening of oils and increase in their viscosity index. They also reduce the solidification point of oils. For obtaining the polymethyl methacrylates the mixtures of alcohols C_{10}-C_{12} and C_{12}-C_{14} can be used. Polymethyl methacrylates tested in the USSR on the experimental scales considerably exceeded the known additives AZNII-1 and AZNII-TsIATIM-1. Thus, for example, transformer oil with the viscosity index of 73 with an additive of 0.25% polymethyl methacrylate had a viscosity index 105, and with 1% - 138. Distillate oil with the solidification point of 10°C with the additive 0.25% polymethyl methacrylate had a solidification point - 31°C (with additive AZNII-1 only - 18°C).

Polymethacrylates are used as protective coatings. For these purposes are suitable the polymethacrylates, prepared into themselves the different organic polar solvents, their which most frequently are used aromatic hydrocarbons, esters and ketones. These solutions are used as the carriers of paints/colors for the heat resistant white enamels (stable to a change in the color) and the enamels, intended for coating of instruments. Varnishes on the basis of

methylmethacrylate possess good adhesive properties and are used for coating of automobiles.

Methylmethacrylate is used for production of methacrylic alkyd resins, which are used for decorative coatings of metal. These resins differ in terms of very rapid solidification in air, uniform distribution on the painted surface, and coatings on their basis - by life and by the capacity to retain color.

Copolymers of methylmethacrylate with styrene recently found wide application. They are used for the production of the corrugated of sheet and other materials, which ensure such properties as weather resistance, transparency and color fastness. Is developed methacrylic syrup, predominantly on the basis of methylmethacrylate, which contains a small quantity of the crosslinking agents. This product is used with the same amplifiers, as in the case of unsaturated polyesters/polyethers. Methacrylic syrup is colorless, it possesses a good adhesion to fiberglass and weather resistance.

In Soviet Union underwent industrial development new class of polymerizable oligomers - polyester acrylates. Polyester acrylates are the reaction products of glycols or polyatomic alcohols, maximum dibasic acids and acids of the acrylic series/number most frequently of methacrylic acid. These oligomeric compounds are a liquid of different viscosity or the readily fusible substances, capable during the introduction of initiators and accelerators at a normal

atmospheric pressure, room or moderately elevated temperature, without the additive of volatile monomers, to be converted to the heat resistant nonmelting and undissolved polymers.

Especially important property of oligomer-polyether/polyesteracrylic is to be polymerized already in articles (coatings) with the formation of products, which possess high heat resistance, chemical and mechanical strength.

Developed polyester-acrylate varnishes and compounds make it possible to considerably intensify technological processes in electromachine building and apparatus construction, in particular, impregnation and drying of electric windings. The duration of these processes is shortened from 20-30 h to several hours and even minutes with a simultaneous improvement in the quality of the produced electrical equipment.

In radio electronics are used heat-resistant sealing compounds on basis of polyester acrylates, sealing materials for semiconductor devices and so forth; in paint and varnish industry application of polyester acrylates makes it possible to reduce consumption of vegetable oils.

Replacement in composition of connecting/cementing for glass-fiber-reinforced plastics toxic monomers and solvents by polyester acrylates gives possibility to prepare large-size articles

at low temperatures and to considerably improve sanitary-hygenic working conditions.

Soviet chemists, who lead investigations in region of acrylates and methacrylates, successfully perform work on their modification somehow:

- innoculative copolymerization of acrylic monomers with polyethylene obtained film coatings, which retain all properties of polyethylene and which additionally acquire exceptionally/exclusively valuable property of polyacrylates - weather resistance.

- innoculative copolymerization of acrylic and methacrylic monomers with protein substances obtained protein-polymer film formers, which match water vapor permeability and heat resistance of initial protein with weather resistance, elasticity and water resistance of acrylates.

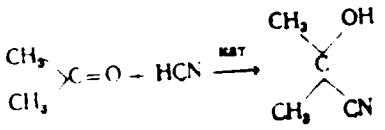
- by chemical inoculation obtained highly elastic materials, suitable for their utilization in the form of films. Obtained by this method organic glasses showed a good fungus resistance and stability under the conditions of the tropical climate.

- by block copolymerization of methylmethacrylate with different organophosphorus compounds there are obtained organic glasses with reduced flammability.

- by copolymerization of methylmethacrylate with organo-tin methacrylates are obtained organic glasses, capable of delaying

X-rays. These glass are more resistant to the effect of different form of emissions and the thermal oxidation.

Industrial methods of synthesis of methacrylates. At present methacrylates are obtained in the industry by continuous method from acetone and hydrocyanic acid through acetone cyanohydrin. The addition/connection to it of hydrocyanic acid with the formation of acetone cyanohydrin is one of the most important reactions of acetone. Reaction easily occurs in the presence of the basic catalysts:



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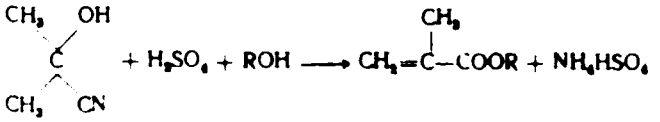
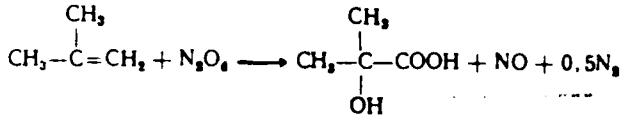
Acetone cyanohydrin can be dehydrogenated for one operation, as a result of which is obtained methacrylic acid or its ethers/esters:



yield of methylmethacrylate is approximately 80%. In the USSR this method was maintained on the industrial scale in the middle of the Thirties.

The method of obtaining methacrylic monomers from isobutylene and technical nitric acid through α-hydroxyisobutyric acid is also developed. Recently α-hydroxy

acids begin to acquire increasing independent importance and as the intermediate products of the synthesis of acrylic and methacrylic derivatives. There is greatest practical interest in the synthesis of α-hydroxyisobutyric acid. Overall reaction of the formation of α-hydroxyisobutyric acid can be written in the following form:



Virtually the expenditure/consumption of nitrogen tetroxide composes 1.25 mole to the mole of isobutylene. In order as the raw material to use not nitrogen tetroxide, but technical nitric acid, nitrogen oxide is oxidized into tetroxide by nitric acid.

Obtained thus nitrogen tetroxide it is completely sufficient for creation of production of α-hydroxyisobutyric acid, using as raw material isobutylene and technical nitric acid.

It was recently established that α-hydroxyisobutyric acid can be used for obtaining of alkyd resins and drying oils for purpose of abbreviation of expenditure/consumption of vegetable oils, and also for synthesis of methacrylic acid. Is developed the technological process, during which on the mixed calcium-phosphate catalyst, which possesses that dehydrating and that hydrolizing with properties, α-hydroxyisobutyric acid easily is dehydrated into the methacrylic with a yield of not less than 90 mol. % to reacted hydroxy acid. Technical and economic estimation showed that the cost/value of the methacrylic monomers, obtained according to the new method, 2-2.5

times lower than cost/value of the monomers, developed at present by the industry through acetone cyanohydrin.

Furthermore, synthesis of methacrylic monomers from isobutylene and nitric acid in comparison with acetonecyanohydrin has series/number of advantages, namely:

- easier creation of continuous process.

- method is based on widely accessible raw material (isobutylene and technical nitric acid).

- there are no production wastes.

- production in principle can be organized in any place, since initial products easily are transported.

- process is not connected with application of such toxic products as hydrocyanic acid and acetone cyanohydrin. In the USSR a very promising method of obtaining the methacrylic acid via the oxidative ammonolysis of isobutylene is also developed.

In connection with this it is not excluded that new methods of obtaining methacrylates on base of isobutylene in the course of time will engage one of leading places in synthesis of methacrylates, but it can be and they will displace classical acetonecyanohydrin method.

For different branches of industry are required poly-acrylate and poly-methacrylic materials, which possess stability at high and low

temperatures, by high mechanical and chemical strength, by elasticity and atmospheric resistance. The creation of such materials on the base of acrylates is our nearest problem. For its successful solution scientific investigations must be conducted in two main trends:

- the improvement of those existing and the development of the new methods of polymerization and copolymerizing the known acrylic monomers.

- application of new acrylic and methacrylic monomers, mainly, which contain different functional or hetero-organic groups.

Both directions must supplement each other. Already at present there are numerous examples of the successful application of monomers with the functional groups in obtaining of poly-acrylate materials. The introduction of functional groups to polymeric and telomeric molecules by copolymerizing the small quantities of monomers, which contain such groups, with the basic monomers makes it possible to change the reaction of the macromolecules of polymer and thereby to add to polymeric material new properties. In connection with this the development of synthesis and the study of the properties of new acrylic and methacrylic monomers with the functional groups are extremely urgent.

Organization in our country of Soviet large-capacity production of polyacrylates and polymethacrylates is proof of the great successes of Soviet chemical industry. The production of these polymeric

materials became possible as a result of introducing the fundamentally new methods of obtaining and polymerization of acrylic and methacrylic monomers.

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